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Sampling of Automobile Interiors for Organic Emissions

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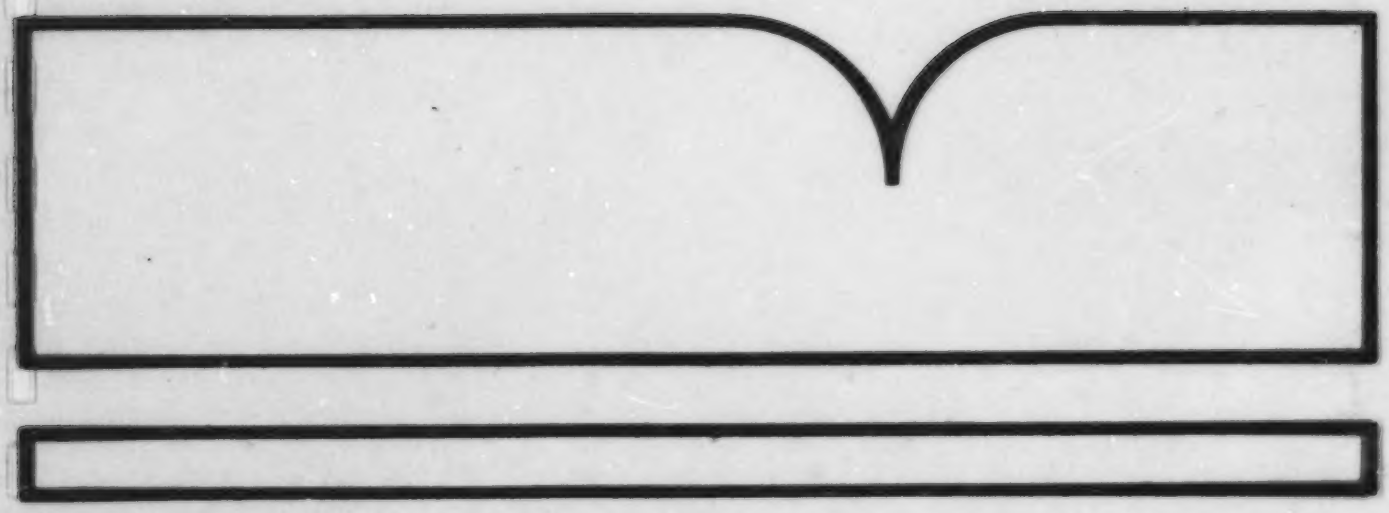

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SAMPLING OF AUTOMOBILE INTERIORS FOR ORGANIC EMISSIONS

by

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16. ABSTRACT

A study was undertaken to determine the concentrations of N-nitrosamines (specifically N-nitrosodimethylamine and N-nitrosomorpholine), hydrogen cyanide, and other volatile organic compounds emitted from the interiors of new cars. The three cars tested were placed in an enclosed shed and heated with infrared tungsten lamps to simulate the temperature rise (heat build-up) in the interior of the cars as if the cars were sitting outdoors on a hot sunny day. The air inside the car as well as the air inside the shed was sampled during the entire temperature rise of "heat build-up" cycle (ambient, ambient to 60°C, and 60°C). These air samples were pulled through cartridges containing adsorbents such as Thermosorb (specifically to collect for N-nitrosamines) and Tenax (used to collect volatile organic compounds), as well as pulling the air through impingers and also into Tedlar bags to analyze for hydrogen cyanide. Low concentrations of N-nitrosodimethylamine and N-nitrosomorpholine were measured in the car interiors during the heating process, but higher concentrations of these same N-nitrosamines were measured outside of the cars. No hydrogen cyanide was detected inside of or outside the two cars tested for this compound. A significant background (mainly gasoline vapors) was obtained for the measurement of other volatile organic compounds, which prevented any meaningful comparison between the car interior and shed emissions. This report covers a period from May, 1980 to February, 1981 and work was completed as of February, 1981.

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This report covers a period from May, 1980 to February, 1981 and work was completed as of February, 1981.

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SECTION 1

INTRODUCTION

Low concentrations of potentially carcinogenic organic vapors such as vinyl chloride, N-nitrosamines, and benzene have previously been identified in the interiors of many cars driven in this country (1,2,3). The emissions of these vapors have been attributed to the materials and adhesives used in the interiors of cars for seating, dash boards and trims. Many different types of plastics, (4,5) rubber (6), vinyl fabrics and adhesives are used in a diversity of manufacturing processes to reduce the costs of producing new cars and to reduce the weight of these cars (for improved gas mileage). It is, therefore, useful to investigate the potentially carcinogenic interior emissions from these cars. The purpose of this study was to identify and quantitate some of these interior emissions in new model cars, especially N-nitrosamines, hydrogen cyanide (HCN), and other volatile organic compounds. Three cars, a 1980 Buick Skylark, a 1980 Mazda GLC, and a 1981 Plymouth Horizon, were placed in an enclosed shed and the interior air of these cars as well as the shed air was sampled before, during and after the cars were heated. Each car was heated to 60°C by focusing six tungsten filament heat lamps on the interior of the car in order to simulate the temperature rise or "heat build-up" inside a car when it is sitting outdoors on a hot sunny day.

The use of an enclosed shed allowed for a controlled environment in order to reduce the potential for outdoor air contamination (7,8).

SECTION 2

CONCLUSIONS

N-nitrosamines were detected in the interior air of all the cars while the cars were heated and at steady state (60°C) temperature. The levels of N-nitrosamines measured inside the autos ranged from an average of <0.04 to 0.37 $\mu g/m^3$ for N-nitrosodimethylamine (NDMA) to an average of $\leq$ 0.06 to 0.25 $\mu g/m^3$ for N-nitrosomorpholine (NMOR); limits of detection were 0.04 $\mu g/m^3$ for NDMA and 0.06 $\mu g/m^3$ for NMOR. The concentrations of these N-nitrosamines measured in the shed, however, were on average two times greater than those measured in the interior.

The cars were subjected to the heating cycle for two days before the usual sampling test cycle was performed (with and without having first purged the interiors with air from outside the shed) in order to determine if an increased amount of these compounds could be produced by "aging" the cars. The N-nitrosamine levels measured under these "drastic" conditions were higher in most cases. The levels of N-nitrosamines measured in the shed, however, were usually greater than those measured in the interior.

Measurements of N-nitrosamines in the trunk of the Buick Skylark were on average one and one-half to two times the concentration measured in the shed. These values, however, were obtained even when the spare tire was removed, indicating that other possible sources of N-nitrosamines must be present.

No HCN was detected in either the interior of the cars tested or in the shed during the entire heating cycle.

The results of the thermal desorption-Gas Chromatographic/Mass Spectrometric (GC/MS) analyses of the tenax cartridges indicated that small quantities of gasoline were still present in the cars even though the gas

tanks were removed and the fuel lines emptied. These gasoline vapors and the vapors of laboratory solvents such as methylene chloride and tetrachloroethylene in high concentrations did not permit evaluation of whether other potential carcinogenic or toxic compounds were being emitted from the interior of the cars tested.

SECTION 3

RECOMMENDATIONS

Further study of a larger, more statistically significant sample is recommended before any conclusions can be made concerning N-nitrosamine emissions in the interiors of new cars.

Instead of purging the shed with air outside the shed, hydrocarbon-free air, nitrogen, or helium should be used as well as removing the carburetor and fuel lines from the cars in order to define clearly the other volatile organic emissions.

Other potential emissions such as vinyl chloride, aldehydes, and ketones should be measured in this controlled environment.

SECTION 4

EXPERIMENTAL PROCEDURES

CARS TESTED

Three cars were tested in this study. These cars were chosen for testing based on their availability. Two of the cars were rented from a local car dealer and the third was obtained from a car rental agency. The characteristics of the test cars are listed in Table 1. To prepare the cars for testing, the gasoline tanks were removed and the cars were operated until the gasoline supply was exhausted.

Table 1. Characteristics of Test Cars

Model	Model Year	Country and date of manufacture	No. of miles of speedometer	Type of interior	Month tested	Tire manufacturer
Skylark 4 door sedan	1980	USA (3/80)	82.3	Tan vinyl	10/80	Goodrich
Mazda 2 door GLC	1980	Japan (4/80)	28.0	Black vinyl	8/80	Bridge-stone
Plymouth Horizon 2 door hatchback	1981	USA (10/80)	1574	Red vinyl trim, red cloth seats	12/80	Fire-

ENCLOSED SHED

The testing and sampling were performed using an enclosed shed (Figure 1), which had been erected for the study of evaporative emissions. The shed is constructed of aluminum walls and floor and has a sheet of Tedlar for the ceiling. The shed has been constructed so that it is "leak tight." Periodically, the shed is checked to insure no leaks have developed by injecting known concentrations of propane and then measuring the total hydrocarbon concentration at one hour intervals for 4 to 5 h using a calibrated total hydrocarbon analyzer. The shed is located in an area of a building that also contains a dynamometer facility and wind tunnel. This building contains many research laboratories that are also involved in programs concerning the collection and identification of air pollutants. There is no physical separation between the test area and the laboratories in this building. The dynamometer was not in operation and no other cars were observed in the testing area at the time of this study.

Tungsten quartz lamps (Fostoria Industries, Inc., Fostoria, Ohio) were used to heat the interiors of the cars. These lamps are the same as those used to dry fresh paint on cars. The heaters consisted of six quartz tungsten tubular infrared lamps contained in a #19 guage steel panel shell finished inside and out with a green baked synthetic enamel. The reflectors were made of a gold porcelain coating. The electrical connections were made using Teflon-insulated, stranded copper wire to the procelain connectors on the lamp housing.

The placement of the lamps is indicated in Figure 2. The distance from the lamps to the roof of the car was approximately 3 ft. At this distance, it required two hours to heat the interiors from ambient temperature to 60°C. Four lamps were focused to heat the roofs of the cars. Two other lamps were focused to heat both the roof and the interior, but were placed opposite to

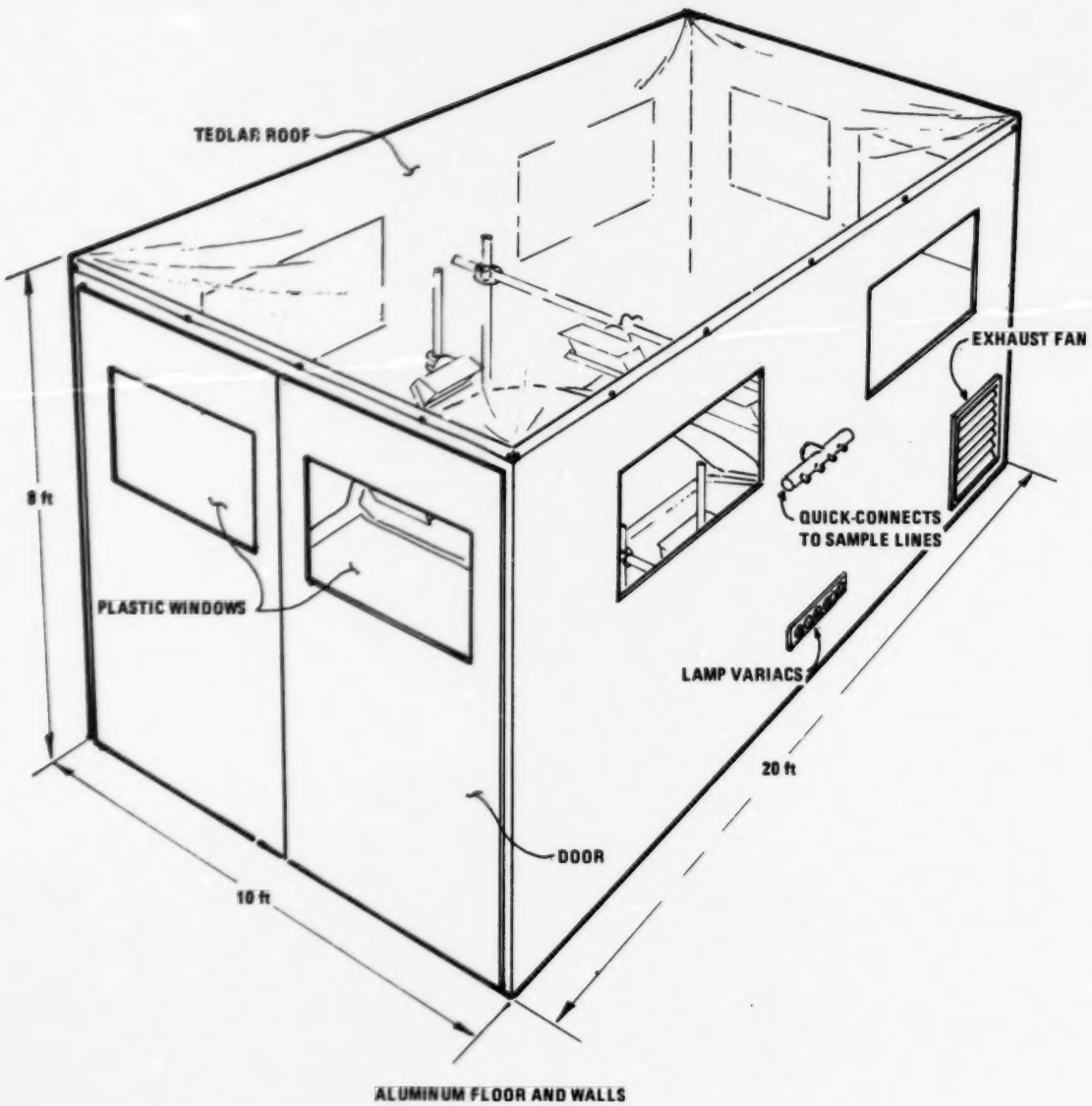

Figure 1. Shed, Horiba, Inc., model EPS76, volume: 1600 ft^3.

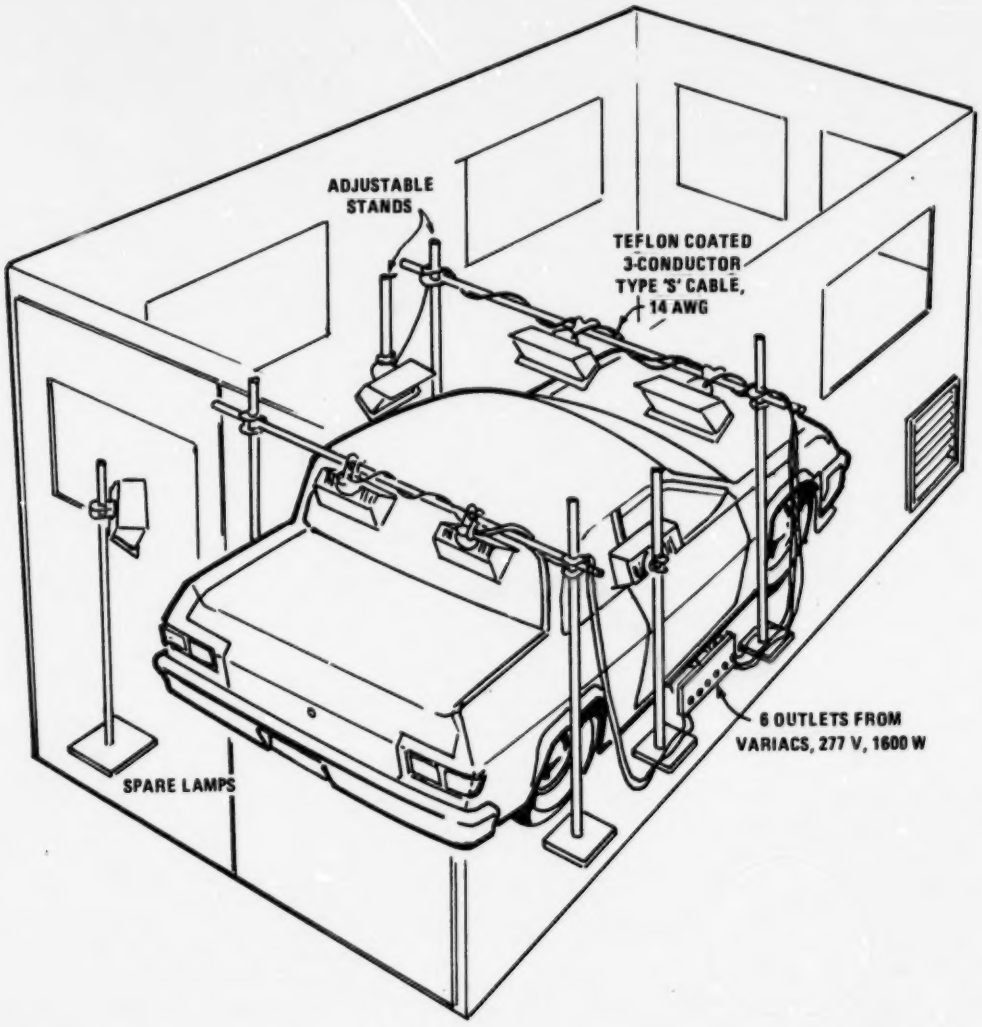

Figure 2. Placement of heating lamps in evaporative shed.

where samples would be collected. The light intensity of each lamp, and therefore the amount of heat produced, was controlled by Variacs outside of the shed.

A shed was constructed outside the building where the actual testing was to be performed in order to determine the expected final interior temperature and the time required to obtain this final temperature for a typical sunny day in the middle of June. The shed consisted of a wood frame covered with Tedlar film that was sealed with Visqueen tape.

The average ambient temperature for the three days measured ranged from (10 to 21°C) at 9:00am and from (30 to 32°C) at 11:00am. The vehicle interior temperatures were measured by a thermobouple positioned in the front seat. With all the vehicle windows closed, interior temperatures ranged from 56 to 64°C. In an afternoon test when the ambient temperature ranged from 29 to 31°C, the vehicle interior temperature ranged from 49 to 61N.D.(2)°C.

TESTING PROCEDURE

The placement of the sampling probes is shown in Figure 3. Thermocouples were also placed at the same locations in which samples were to be collected in order to monitor the temperatures at each particular sampling location. The sampling was performed at three different temperatures. The first sample was collected at ambient temperature, the second was collected during the heat build up and the third was collected one half hour after the temperature in the interior stabilized at 60°C. Some samples were also collected in the trunk of the Skylark with and without the spare tire.

The "worst case" test situation was done by performing the "heat build up" cycle with the cars in the shed for two days without sampling and allowing

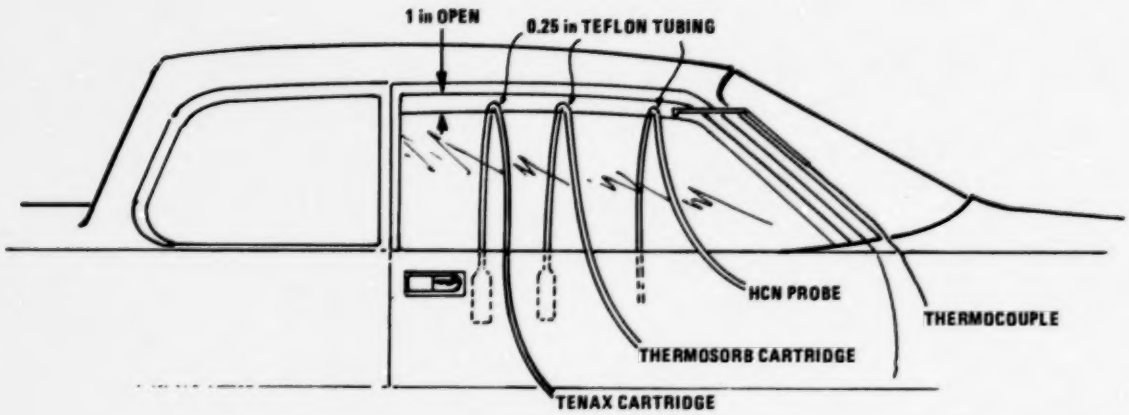

Figure 3. Passenger window showing placement of sample probes.

the cars to remain in the shed for the two days. On the third day the air was sampled, with and without having first purged the car interior or shed air, following the usual "heat build up" procedure. The cartridges used for sampling and analysis of the volatile organic compounds and nitrosamines were placed inside the cars. Preliminary tests had indicated that at an interior temperature of 60°C there was no difference between collecting samples in cartridges placed inside the car and collecting samples from the interior through 1/4 in. Teflon tubing with the sampling cartridges placed outside the shed.

After ambient temperature sampling was completed, these cartridges were removed from the shed. When the sampling during the "heat build up" was completed, the pumps were shut off for these cartridges. They were removed after the 60°C sampling test had also been completed. The trunk of N.D.(2)the Skylark was sampled with and without the spare tire during this study.

Before the testing was initiated, the shed (with no car inside) was heated to 60°C for an entire day and then allowed to cool to ambient overnight. This procedure was repeated for several days. The shed was then flushed each day with room air. The day prior to testing, background samples from the shed (with no car inside) were collected at ambient, ambient to 60°C, and 60°C.

A Thermosorb sampling system was used to collect for potential N-nitrosamine emissions as this system has been demonstrated as the method of choice for retaining 100% of any pre-loaded N-nitrosamines (9,10). The Thermsorb cartridges used to collect the air samples were supplied and analyzed by the Thermo Electron Corp. (Waltham, Mass.). Air samples were collected at a flow rate of 1 l/min using calibrated flowmeters.

Two completely separate methods were used to measure HCN: one method involved collecting the air in a Tedlar bag and then analyzing a sample of this bag using a GC coupled to a chemiluminescent detector (11), the other method involved collecting the air sample into an impinger containing 25ml of 0.625N NaOH and analyzing the samples using a colorimetric chemical analysis system (12). The limit of detection for HCN by the GC method is 10 ppb and is 25 ppb for the colorimetric method. These limits of detection were determined by Stump (13) when he compared and adapted three different methods (1- GC-chemiluminescent detector system (11), 2- colorimetric method(12), and 3- The use of an optical gas filter correlation spectrometer) to analyze for HCN in automobile exhaust.

Tenax cartridges were prepared as described elsewhere(1). The air samples collected in the Tenax cartridges were analyzed using a Nutech Model 320 thermal desorption system coupled to an H.P. Model 5992A gas chromatograph/mass spectrometer (GC/MS) system. The sampling rate for the collection of air through the Tenax cartridge was 300 ml/min, as measured by calibrated flow meters.

GC/MS PARAMETERS

The GC column was a 25 m by 0.32 i.d. SP 2000 fused silica capillary column (Hewlett Packard). Helium was used as the carrier gas and it was controlled at 3 ml/min using a Tylon flow controller. The GC temperature programming parameters were as follows: initial temperature, 30°C; initial time, 8.0 min; program rate, 4°C/min; final temperature, 150°C; and final time, 5.0 min. The mass spectrometer parameters were as follows: scan speed, 690 amu/s; ionizing voltage, 70 eV; and electron multiplier, 2600 V. The Nutech desorption unit parameters were as follows: cartridge desorption

temperature, 240°C; cartridge desoprtion time, 5.0 min; helium flow through cartridge, 15 ml/min; cold trap temperature, -150°C; and final temperature of that trap, 190°C.

SECTION 5

RESULTS AND DISCUSSION

The results of the analyses for the N-nitrosamines (NDMA and NMOR) are listed in Tables 2 and 3. No NDMA or NMOR was detected in the shed (without the car inside), at ambient, ambient to 60°C, or at 60°C, or in the air surrounding the shed. Also, there was no hydrogen cyanide detected in these background samples.

In general, the results indicate the NDMA and NMOR were emitted at low concentrations when the interiors of these cars were heated from ambient to 60°C. In almost all cases, greater quantities of NDMA and NMOR were measured in the shed air than in the cars' interior during every test cycle.

At ambient only NDMA was detected in the interior of the Plymouth Horizon. At 60°C NDMA was detected and measured in all of the car interiors and only in the Mazda was NMOR not detected. The average interior concentration of NDMA and NMOR emitted at 60°C were lower, 0.17 $\mu g/m^3$ NDMA and <0.13 $\mu g/m^3$ NMOR, than the levels measured in another study (2), 0.3 $\mu g/m^3$ NDMA and 0.67 $\mu g/m^3$ NMOR.

The NDMA emitted from the interior of the Plymouth Horizon increased from trace levels to 0.55 $\mu g/m^3$ for the three times the car was heated. No NMOR was detected during this experiment. Only one sample of the interior of the Skylark and Mazda was obtained during the "heat build up" so that no determination could be made of an increase or decrease in NDMA emission over time. The data from the "aging" testing indicates, however, that little if any NDMA was produced during the two times the cars were heated.

Table 2. Normal Test Cycle Analyses for NDMA and NMOR

Car (condition)[c,d]	NDMA[a] ($\mu g/m^3$)			NMOR[b] ($\mu g/m^3$)		
	Interior	Shed	Trunk	Interior	Shed	Trunk
Skylark						
Ambient temperature[e]	N.D.[f](2)[g]	0.18(2)	0.15(2)	0.14(2)	N.D.(2)	N.D.(2)
Heat build up	0.37(1)	0.16(1)	N.D.(1)	N.D.(1)	N.D.(1)	N.D.(1)
60°C	0.18(3)	0.48(3)	0.54(3)	0.25(3)	0.42(3)	0.39(3)
Mazda						
Ambient temperature	N.D.(3)	tr(3)[h]	N.R.[i]	N.D.(3)	N.D.(3)	N.R.
Heat build up	tr(1)	0.21(1)	N.R.	N.D.(1)	N.D.(1)	N.R.
60°C	0.18(3)	0.40(3)	N.R.	N.D.(3)	tr(3)	N.R.
Horizon						
Ambient temperature	0.16(3)	N.D.(1)	N.R.	N.D.(3)	N.D.(1)	N.R.
Heat build up	0.30(3)	0.13(1)	N.R.	N.D.(3)	N.D.(3)	N.R.
60°C	0.16(4)	0.61(4)	N.R.	N.D.(3)	0.19(4)	N.R.

[a] $L_{LD} = 0.04\ \mu g/m^3$

[b] $L_{LD} = 0.06\ \mu g/m^3$

[c] Trunk temperature was 46°C when interior temperature was 60°C

[d] Duration of experiments: ambient temperature, one hour; heat build, two hours; 60°C, one hour

[e] Car initially equilibrated in shed over weekend; average of one sample obtained with and one sample obtained without purging of car and shed

[f] N.D. = not detected

[g] Number in parentheses is number of times experiment was run

[h] N.R. = no sample collected.

[i] Trace value < 3 x L_{LD} NDMA < 0.12 $\mu g/m^3$; NMOR < 0.18 $\mu g/m^3$

Table 3. Analyses for NDMA and NMOR After Two Days of Heat Build Up

Car (condition)[c,d]	NDMA[a] ($\mu g/m^3$)			NMOR[b] ($\mu g/m^3$)		
	Interior	Shed	Trunk	Interior	Shed	Trunk
Skylark						
Ambient temperature[e]	N.D.[f] (2)[g]	0.15(2)	0.18(2)	N.D.(2)	N.D.(2)	N.D.(2)
Heat build up	N.D.(2)	0.19(2)	0.38(2)	N.D.(2)	N.D.(2)	0.23(2)
60°C	0.33(4)	0.36(2)	0.64(2)	0.34(4)	0.42(2)	0.60(2)
Mazda						
Ambient temperature	N.D.(3)	N.D.(3)	N.D.[h]	N.D.(3)	N.D.(3)	N.R.
Heat build up	tr(2)[i]	0.45(2)	N.R.	N.D.(2)	0.22(2)	N.R.
60°C	0.22(2)	0.51(2)	N.R.	N.D.(2)	0.32(2)	N.R.

a $L_{LD} = 0.04\ \mu g/m^3$

b $L_{LD} = 0.06\ \mu g/m^3$

c Trunk temperature was 46°C when interior temperature was 60°C

d Duration of experiments: ambient temperature, one hour; heat build, two hours; 60°C, one hour

e Samples collected with and without spare tire. After two days heat build up, car and shed were not purged

f N.D. = not detected

g Number in parentheses is number of times experiment was run

h N.R. = no sample collected.

i Trace value < 3 x L_{LD} NDMA < 0.12 $\mu g/m^3$; NMOR < 0.18 $\mu g/m^3$

The individual N-nitrosamine concentrations measured from the air collected inside the trunk of the Skylark were greater than the concentrations measured either in the interior of the car or the shed for almost all the tests. The spare tire of the Skylark was removed before the second "aging" test was performed because the spare tires have been suspected as a source of N-nitrosamines (6). The levels of N-nitrosamines measured after this test, however, were not significantly different than the values obtained when the spare tire was in place. The trunk of the Skylark has a physical barrier separating it from the interior; the Plymouth Horizon and Mazda are hatchbacks. The individual levels of N-nitrosamines detected in the interior of the Skylark, however, were relatively higher in most of the test cycles than the levels measured in the other two cars.

The results of the worst case "aging" tests did not appear different than any of the other tests, indicating a steady-state emission of N-nitrosamines even when the interiors of these cars were heated and were not purged.

The HCN collection and analyses were performed on the Skylark and Mazda. No HCN was determined to have been emitted from the interiors of these two cars. The results indicate that even relatively high (60°C) temperature and "worst case" conditions such as baking out the car for two days without purging the interior did not produce any detectable HCN.

The analyses of the shed and car interior air samples collected on Tenax cartridges were performed later in the testing program. The cartridges were stored in a -20°C freezer until analyzed. The subsequent analyses of many of these cartridges indicated that the sampling time could have been reduced to approximately one quarter of the actual sampling rate because overloading of the column was observed on the total ion chromatogram.

The results of the thermal desoprtion - GC/MS analyses of these Tenax cartridges did indicate that low quantities of gasoline vapors were still present in the cars even though the gas tanks were removed and the fuel lines were emptied. Benzene and toluene were usually the compounds of greatest abundance in the total ion chromatograms. The areas (total abundance) of the other peaks associated with the components of gasoline increased as the cars were heated which further indicates that these vapors were present in the cars.

The freons and laboratory solvents such as methylene chloride, 1,1,1 trichloroethylene and carbon tetrachloride were also detected in varying concentrations throughout this study. Tetrachloroethylene had the greatest abundance when a background sample of the shed (with no car inside) was collected and analyzed. This compound was also detected in varying concentrations in all the other samples analyzed.

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